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(71)Applicant : TOYO INK MFG CO LTD

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(72)Inventor : TOMIHARI TOSHIYA
SATO SHINICHI

(54) SURFACE-TREATED CARBON BLACK, PRODUCTION THEREOF, AND DISPERSION CONTAINING THE SAME

(57)Abstract:

PROBLEM TO BE SOLVED: To obtain a surface-treated carbon black which can be homogeneously dispersed by treating the surface of a carbon black with an amine compd. having a prim-amino group.

SOLUTION: The surface of a carbon black is treated with a surface-treating agent represented by the formula: $H_2N-R'-(NR_2R_3)_n$ (wherein R_1 is a linking group comprising a chemically reasonable combination of at least two kinds of atoms selected from among carbon, hydrogen, oxygen, and nitrogen atoms and having a mol.wt. of 14-1,000; R_2 and R_3 are each independently a 1-18C linear or branched, optionally substd. alkyl group provided R_2 and R_3 may combine with each other, optionally together with a nitrogen or oxygen atom, to form a 5- or 6-membered ring optionally further substd.; and n is 1 or 2). Pref., R_1 is a 1-4C alkylene; and R_2 and R_3 are each independently a 1-4C alkyl.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[Industrial Application] This invention relates to the dispersing element which comes to use the carbon black by which surface preparation was carried out, its manufacture approach, and it.

[Description of the Prior Art] Carbon black is widely used in fields, such as printing ink, a coating, and a plastics formation ingredient, as a color pigment or a protection-from-light ingredient. In order to raise coloring nature and the light absorption engine performance in these products, homogeneity must be made to distribute carbon black in a solvent or resin, but since dispersibility of carbon black is not good, in order to obtain homogeneity distribution, great energy is needed in respect of a process. Generally the approach of adding various kinds of dispersants as a means to ease such a problem is performed. However, even if it used this approach, since the dispersibility satisfied enough is not only hard to be acquired, but the degradation of ink or a coating was caused when the compatibility of a dispersant and a resinous principle was bad, according to the dispersant, the resinous principle had to be chosen, or the dispersant had to be chosen according to the resinous principle, and there was a problem that versatility was missing. It sticks to the carbon black in which the relation of the surface state of carbon black and resin to distribute generally has a basic front face (electron donor nature) strongly with a giant molecule with an acid functional group, and there is an inclination to be easy for a giant molecule with a basic functional group to adsorb in the carbon black which has an acid front face (electron acceptor nature). Therefore, when the charge polarity of the carbon black and resin to mix is different, good distributed stability will be acquired. In order to raise the dispersibility of carbon black using such a property, without using a dispersant, the approach of reforming the polarity on the front face of a particle is proposed for many years. However, if it is in the ink and coating industry which uses the various pigments with which surface charge differs, and an additive, since it is difficult to control the surface charge of all coatings, in order to acquire sufficient distributed stability, it has not yet resulted. In order to solve these problems, many approaches of mixing or distributing carbon black to homogeneity are examined by covering a carbon black front face with various kinds of surfactants and resin, and heightening an affinity with the shape of a solid-state, and a liquefied base material. However, the present condition is that the result which the carbon black which the front face obtained by the old approach comes to cover is not improved at the degree which the compatibility with other matter expected, but is not necessarily satisfied is not obtained. Moreover, the attempt which improves the dispersibility of carbon black is made by oxidizing a carbon black front face and introducing an acid functional group. The liquid-phase-oxidation method using vapor-phase-oxidation methods, such as ozonization and plasma treatment, a nitric acid, hydrogen peroxide solution, sodium perchlorate, etc. as the oxidation-treatment approach is devised. When general oxidation-treatment conditions are compared, the liquid phase oxidation of effectiveness is higher than vapor phase oxidation, surface acidity increases [the direction of a nitric acid] from hydrogen peroxide solution, and it is known for liquid phase oxidation that dispersibility will improve. However, in liquid phase oxidation, a mutagen student's problem is pointed out about that the purification after processing is difficult, and the carbon black obtained especially by oxidation by nitric acid. Moreover, in vapor phase oxidation, although there

is an advantage that a purification process is unnecessary, since acidity is low, it has the fault that sufficient dispersibility is not acquired, as point **.

[Problem(s) to be Solved by the Invention] This invention is made in view of the above-mentioned situation, and is for the purpose to offer the carbon black dispersing element containing the surface-preparation carbon black and it from which uniform dispersibility is acquired.

[Means for Solving the Problem] That is, this invention is surface-preparation carbon black characterized by processing a carbon black front face by the finishing agent expressed with the following general formula (1).



The joint radical of the molecular weight 14-1000 which consists of two or more sorts as which R1 is chosen from a carbon atom, a hydrogen atom, an oxygen atom, or a nitrogen atom among [type which consists of a rational combination chemically, R2 and R3 are the alkyl group (R2 and R3 may be united, they may also contain a new nitrogen atom or an oxygen atom, and may form five membered-rings or six membered-rings which may have the further substituent.) which may have the shape of a straight chain and the branched-chain substituent of the carbon atomic numbers 1-18 independently, respectively. n expresses 1 or 2.] Furthermore, this invention is surface-preparation carbon black given [above-mentioned] in invention characterized by the total amounts of surface functional groups of the carbon black before surface preparation being more than 0.2 milliequivalent / g. furthermore, the above-mentioned invention to which, as for this invention, R1 in a general formula (1) considers that the alkylene group of the carbon atomic numbers 1-4, and R2 and R3 are the alkyl groups of the carbon atomic numbers 1-4 independently, respectively as the description -- it is surface-preparation carbon black of a publication either. furthermore, this invention -- the above-mentioned invention -- it is a carbon black dispersing element containing the surface-preparation carbon black of a publication either. Furthermore, this invention is a carbon black dispersing element given [above-mentioned] in invention which comes to distribute surface-preparation carbon black in the resin which has an acid functional group. Furthermore, this invention is the manufacture approach of the surface-preparation carbon black characterized by carrying out surface preparation, carrying out differential powder of the carbon black in the solution containing the finishing agent expressed with a general formula (1) according to the shearing stress by the grinder or the disperser, or the impulse force by media. Furthermore, this invention is the manufacture approach of the surface-preparation carbon black given [above-mentioned] in invention characterized by in process or heat-treating at 40 degrees C - 200 degrees C before and after that of carrying out differential powder of the carbon black into the solution containing the finishing agent expressed with a general formula (1).

[Embodiment of the Invention] This invention is explained to a detail below. Especially as carbon black used for this invention, it is restrained by neither a class nor manufacture hysteresis, and various kinds of things, such as commercial oil furnace black, gas furnace black, thermal black, acetylene black, and channel black, can be used. Moreover, the ozonization usually performed, plasma treatment, and the carbon black by which liquid-phase-oxidation processing was carried out may be used. Generally as for carbon black, functional groups, such as a carboxyl group (-COOH), a phenolic hydroxyl group (- OH), and a quinone radical (>C=O), are generated by the particle front face in a generation process. About the amount of carboxyl groups (-COOH), and the amount of phenolic hydroxyl groups (- OH), it comes [amount / of quinone radicals (>C=O) / a titrimetric method and] out of the quantum of the functional group of these carbon black to measure with the hydrogen absorption process of sodium borohydride, respectively. It is 0.2 - 8.0 milliequivalent /g preferably, the whole quantity of surface functional groups, i.e., total amount, of these surface functional groups, and the carbon black used for this invention is 1.0 - 6.0 milliequivalent /g especially preferably. If there are few total amounts of surface functional groups than 0.2 milliequivalent, there are few adsorption or the reacting points of a finishing agent, and they cannot acquire sufficient surface treatment effectiveness. Although there is especially no problem on the other hand even if it uses carbon black with more total amounts of functional groups than 8.0 milliequivalent / g, improvement remarkable in the surface treatment effectiveness is not accepted. Moreover, although there is especially no limit about the ratio of each functional group, the direction

with many rates of a carboxyl group is desirable. Although the particle size of the carbon black used for this invention is the same as the size range of the carbon black used for usual ink and a usual coating, 0.01-10 micrometers is 0.01-0.5 micrometers especially preferably. However, the particle size said here expresses the first [an average of] particle size measured with the electron microscope used for generally the physical property of carbon black being shown. The finishing agent in this invention is shown by the general formula (1).

General formula (1)

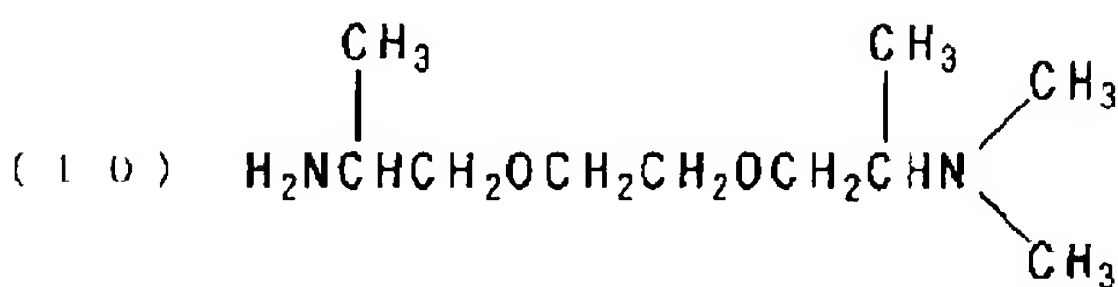
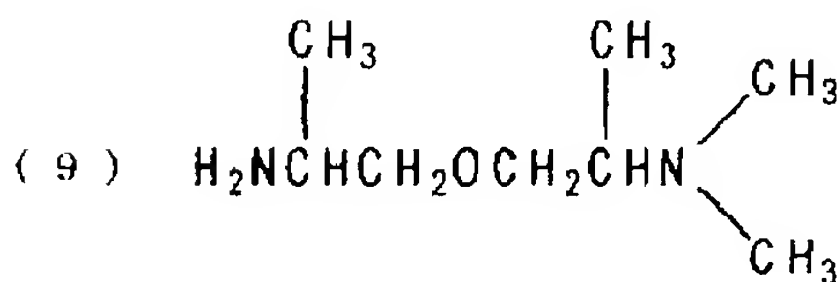
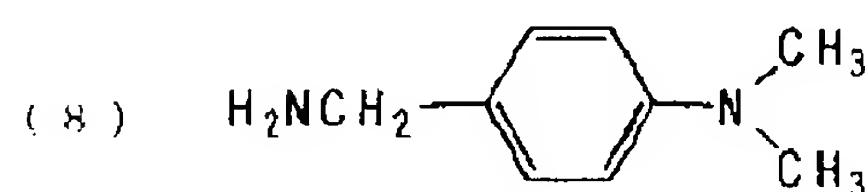
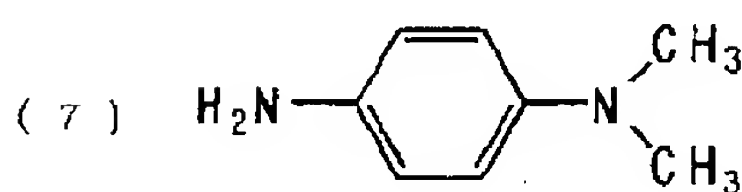
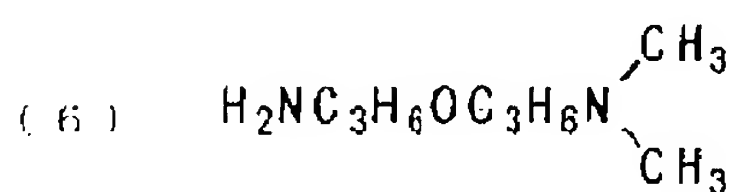
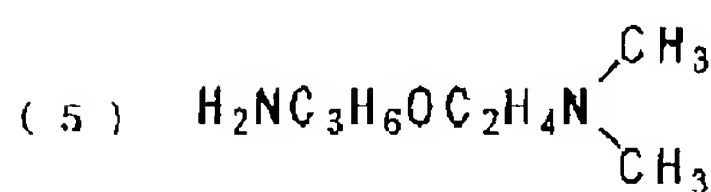
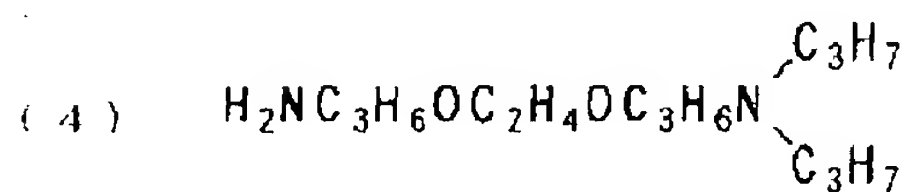
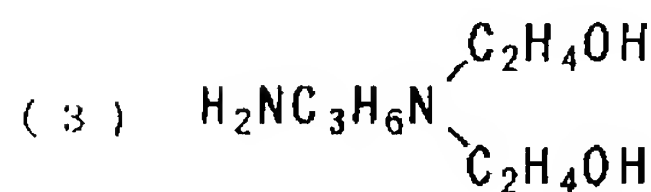
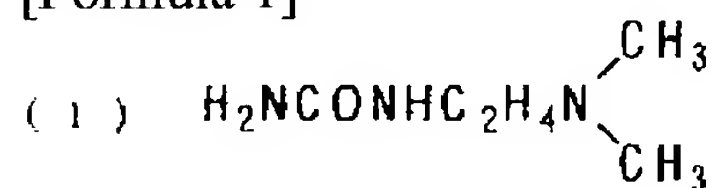


The joint radical of the molecular weight 14-1000 which consists of two or more sorts as which R₁ is chosen from a carbon atom, a hydrogen atom, an oxygen atom, or a nitrogen atom among [type which consists of a rational combination chemically, R₂ and R₃ are the alkyl group (R₂ and R₃ may be united, they may also contain a new nitrogen atom or an oxygen atom, and may form five membered-rings or six membered-rings which may have the further substituent.) which may have the shape of a straight chain and the branched-chain substituent of the carbon atomic numbers 1-18 independently, respectively. n expresses 1 or 2.]

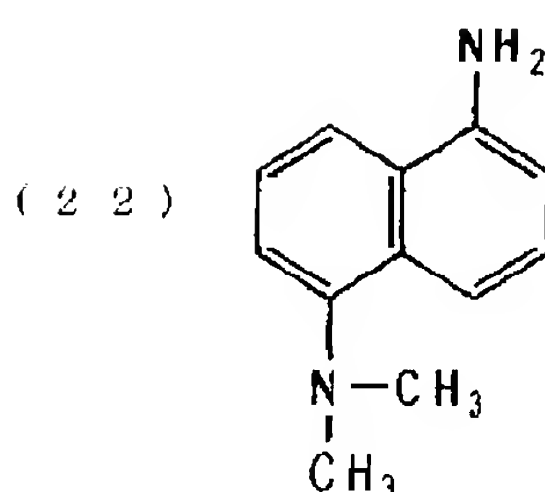
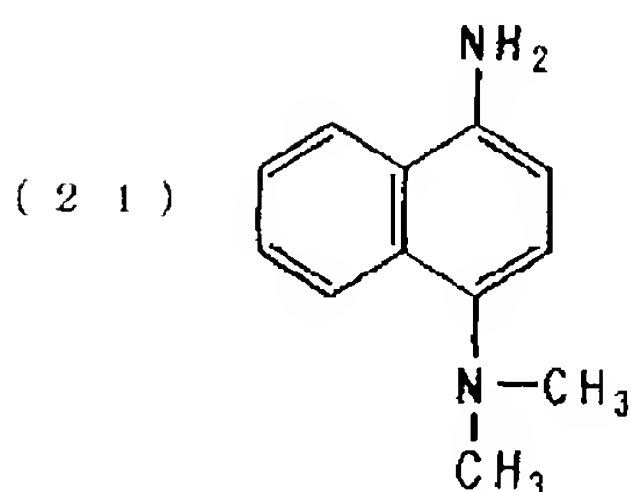
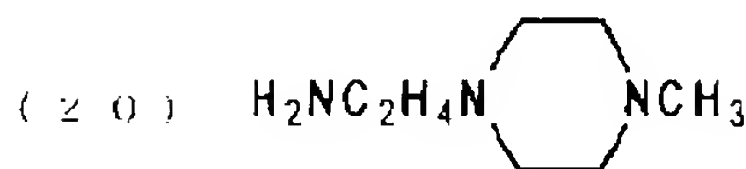
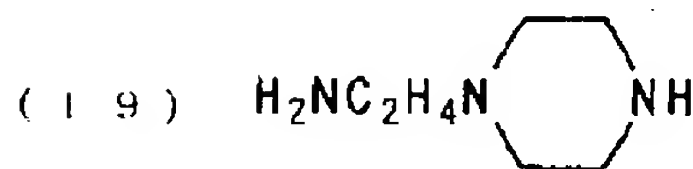
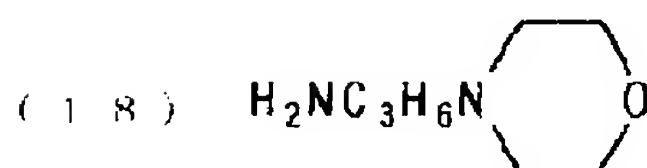
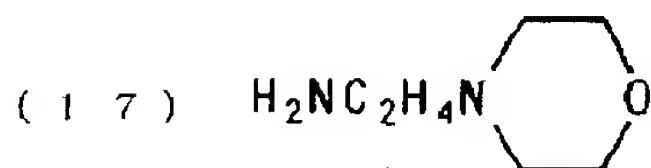
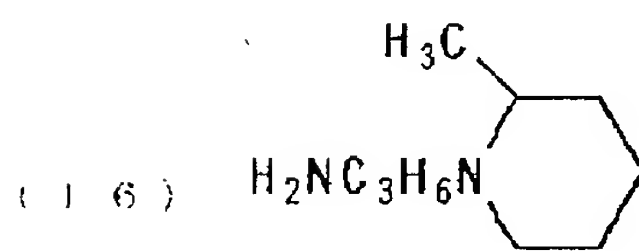
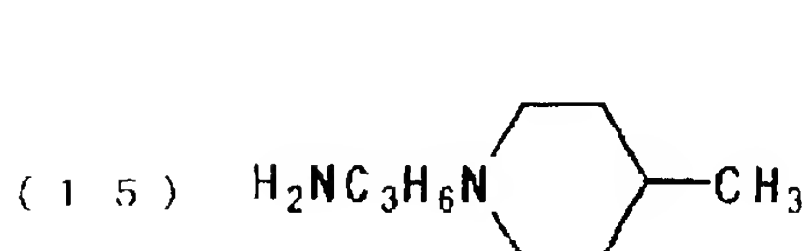
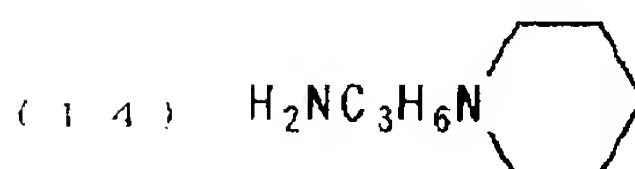
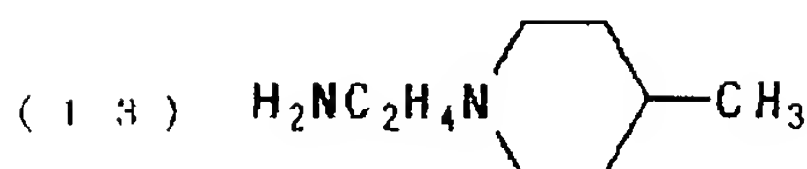
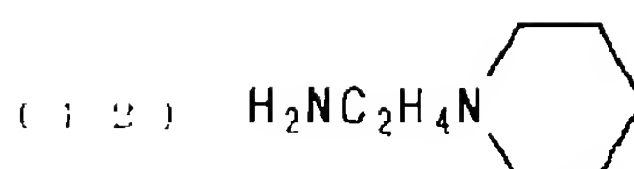
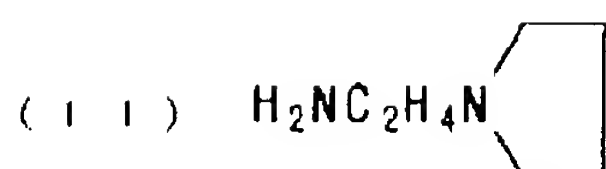
The finishing agent used for this invention shown by the general formula (1) is an amino compound which has the 1st class amino group, it is known that will stick to these amino groups by the acid-base interaction, and they will form a quinone radical and a Schiff base (>C=N-) especially about the 1st class amino group to the carboxyl group and phenolic hydroxyl group which show acidity among the surface functional groups of carbon black in addition to an acid-base interaction, and the high surface treatment effectiveness is acquired according to such an operation. R₁ in said general formula (1) used for the finishing agent of this invention It is the joint radical which consists of two or more sorts chosen from a carbon atom, a hydrogen atom, an oxygen atom, or a nitrogen atom and which consists of a rational combination chemically. For example, the shape of a straight chain, the branched-chain alkylene group of ethylene, a propylene, a butylene, and other resemblance [hexylene and], It is the alkylene group and naphthylene radical containing ethylene oxide, an alkylene group including the ether linkage of propylene oxide and other resemblance, an alkylene group including amide association (formula-CO-NH- or -NH-CO-), a phenylene group, and a phenylene group. These alkylene groups, the phenylene group, and the naphthylene radical may be permuted by the substituent chosen from the group which consists of the alkyl group (for example, a methyl group or an ethyl group) of the carbon atomic numbers 1-4, the alkoxy group (for example, methoxy group) of the carbon atomic numbers 1-4, a hydroxy group, a sulfonic group, and a carboxy group. R₂ and R₃ in said general formula (1) which are used for the finishing agent of this invention are a substituent which has the carbon atom of 1-18 independently respectively. As an example of R₂ and R₃ alkylation radical, a methyl group, an ethyl group, a propyl group, an isopropyl group, butyl, an isobutyl radical, a hexyl group, an octyl radical, a nonyl radical, a decyl group, a lauryl radical, a stearyl radical and the shape of a straight chain similar in addition to this, and a branched-chain substituent are mentioned, for example. For example, a pyrrolidinyl radical, a piperidino radical, and a morpholino radical are mentioned as an example of the heterocycle radical with which R₂ and R₃ were united. These substituents may be permuted by the substituent chosen from the group which consists of the alkyl group (for example, a methyl group or an ethyl group) of the carbon atomic numbers 1-4, the alkoxy group (for example, methoxy group) of the carbon atomic numbers 1-4, a hydroxy group, a sulfonic group, and a carboxy group. Moreover, as for especially R₁, in said general formula (1), it is good that it is the alkylene group of the carbon atomic numbers 1-4, and, as for R₂ and R₃, it is still better respectively that it is the alkylation radical of the carbon atomic numbers 1-4 independently. As an example of the finishing agent used by this invention Although there will be especially no limitation if it is a compound applicable to the structure shown in a general formula (1) For example, N and N-dimethylamino ethylamine, N, and N-dimethylamino propylamine, N and N-dimethylamino butylamine, N, and N-diethylamino ethylamine, N and N-diethylamino propylamine, N, and N-diethylamino butylamine, N and N-dipropylamino butylamine, N, and N-diisopropylamino propylamine, N and N-dibutylamino ethylamine, N, and N-dibutylaminopropylamine, An N and N-dibutylamino butylamine, N, and N-diisobutyl aminopropyl

amine, An N and N-diethylamino hexylamine, N, and N-methyl-lauryl aminopropyl amine, N,N-dioctylamino ethylamine, N, and N-distearyl amino butylamine, N, and N-dimethyl neopentane diamine or the compound shown in No.1-22 of the following is mentioned, you may independent-use it or these may be used together.

[Formula 1]



[Formula 2]



Also in these compounds, N and N-dimethylamino ethylamine, N, and N-dimethylamino propylamine, N and N-dimethylamino butylamine, N, and N-diethylamino ethylamine, N and N-diethylamino propylamine, N, and N-diethylamino butylamine, N and N-dipropylamino butylamine, N, and N-diisopropylamino propylamine, Since dispersibility is excellent, an N and N-dibutylamino ethylamine, N, and N-dibutylaminopropylamine, N, and N-dibutylamino butylamine, N, and N-diisobutyl aminopropyl amine can be especially used for the finishing agent of this invention preferably. The additions of the finishing agent of this invention are 0.01 to 10.0 millimol /g to carbon black. If there are few additions of a finishing agent than 0.01 millimols / g, the dispersion effect of surface-preparation carbon black is not small desirable. Moreover, even if there are more additions of a finishing agent than 10.0 millimols / g, improvement in the addition effectiveness is not accepted. The surface-preparation carbon black in this invention is obtained by carrying out mixed churning of carbon black and the above finishing agents. [Although there is especially no limit in the approach of mixed churning which can be set like a companion, it is desirable to perform surface treatment, carrying out differential powder of the carbon black in the solvent containing a finishing agent according to the shearing stress by the grinder or the disperser, or the impulse force by media.] As the grinder used in this process, or a disperser 3 roll

mills and 2 roll mills to which differential powder of the carbon black particle is carried out with shearing stress, The ball mill to which differential powder of the carbon black particle is carried out according to the impulse force of media, such as a glass bead and zirconia beads, Attritor, a sand mill, KOBORUMIRU, a basket mill, a vibration mill, A paint conditioner, etc. and shearing stress, cavitation, DISUPA which carries out differential powder with a moving vane which is made to generate the collision force, a potential core, etc., The kneader who does differential powder according to the collision force and shearing force of a homogenizer, a KUREA mix (R), etc. carbon black particles or a carbon black particle and a vehicle, or a distributed body wall side, Although an extruder, a jet mill, etc. the ultrasonic disperser that carries out differential powder with a supersonic wave can be mentioned, it is not limited to this. When surface preparation is performed without distributing carbon black, a surface treatment effect can be given even to a more detailed particle front face by performing surface preparation, carrying out differential powder of the floc of a carbon black particle by the grinder or the disperser to the ability not to give a treatment effect only to the floc front face of a carbon black particle. Moreover, it is still more desirable to heat-treat in the stroke which sets like a companion and carries out differential powder of the carbon black, and before and after that. Although the temperature of heat-treatment changes with the solvents and finishing agents to be used, it is desirable to carry out at 40 degrees C - 200 degrees C. When heat-treatment temperature is lower than 40 degrees C, the adsorption or the reaction of a finishing agent to a carbon black particle front face does not advance enough, and the satisfactory surface treatment effectiveness is not acquired. Moreover, even if it makes heat-treatment temperature higher than 200 degrees C, deterioration of about [that improvement in a treatment effect is not accepted], a finishing agent, etc. and the administration energy to a process increase, and it is not desirable practically. Moreover, although refining and washing with a solvent is desirable as for the finishing agent of the surplus contained in the carbon black which processing ended, it is also possible to use it, without removing. The surface-preparation carbon black of this invention is in the condition in which differential powder was carried out by the grinder and the disperser, when the amino group of a finishing agent sticks to the carboxyl group and phenolic hydroxyl group of a carbon black particle front face by the acid-base interaction or forms a quinone radical and a Schiff base in them, the amino group of a finishing agent configures it on a carbon black particle front face, its compatibility with resin and the solvent which are used in case a dispersing element is obtained for this reason improves, and it is thought that the dispersibility of carbon black can be raised. If water, organic solvents, and those mixed liquor are pointed out and a finishing agent is dissolved as a solvent used in the production process of the surface-preparation carbon black of this invention, there will be especially no limit, but in order that ketones may form the amino group and Schiff base ($>C=N-$) in a finishing agent and may check the reaction of the quinone radical on the front face of carbon black, and the amino group in a finishing agent, they are not desirable. Moreover, in a reaction, a well-known catalyst may be used conventionally. polyoxyalkylene resin, polysiloxane resin, polyurethane resin, Pori allylic resin, acrylic resin, vinyl system resin, styrene resin, phenoxy resin, an epoxy resin, fibrin system resin, polyester resin, amino resin, etc. can be used, and independent [in these] as a class of polymer used in case the carbon black dispersing element in this invention is obtained, -- or two or more sorts can be mixed and it can use. Moreover, as an acid functional group, a carboxyl group, a sulfonic group, a phosphoric-acid radical, a boric-acid radical, etc. can be illustrated. It is thought that these acidity functional group can raise compatibility with the carbon black to which the dispersant adsorbed or reacted, and can raise the dispersibility of carbon black. Moreover, in order to raise distributed stability etc. further, a known surface active agent etc. may be used together and lubricant, a leveling agent, a defoaming agent, antiseptics, etc. may be added. There is especially no limit as preparation of the carbon dispersing element using the surface-preparation carbon black obtained by this invention, and the various dispersers or grinder illustrated by the production process of the above-mentioned surface-preparation carbon black can be used. It does not interfere, even if it adds other components after the time of mixing, or distribution. As such a component, they are a binder component, a dispersant, lubricant, an activator, a cross linking agent, a leveling agent, a defoaming agent, a solvent, etc. As a field of the invention of the surface-preparation carbon black in this invention, although there is especially no limitation, it can offer

stability and a uniform dispersing element in the field as which protection-from-light nature, conductivity, endurance, Japanese lacquer malanism, etc. are required, for example, rotogravure ink, offset ink, the back coat for magnetic-recording media, an electrostatic toner, an ink jet, the coating for automobiles, fiber, a plastics formation ingredient, etc.

[Example] This invention is explained based on an example. The "section" is the "weight section" among an example. It is the following, and each functional group of the front face of carbon black A-C used for manufacture of the surface-preparation carbon black of this invention was made and measured, it was totaled, the total amount of surface functional groups was calculated, and the result was shown in Table 1.

(1) Carry out weighing capacity of the measurement desiccation carbon black 1g of the amount of carboxyl groups (-COOH) per 1mg, filter, extract 20ml of supernatants of filtrate, and titrate in the hydrochloric-acid water solution of the centinormal, after shaking 50ml of sodium-hydrogencarbonate water solutions of a decinormal for 4 hours and making them react. The amount of carboxyl groups is calculated by the bottom type as milliequivalent in carbon black 1g (meq./g).

The amount of carboxyl groups = $(50/20 \times 0.01 \times (\text{a titration value} - \text{sky titration value}))$ weighing capacity of the measurement desiccation carbon black 1g of the amount of /carbon black sample weight (2) hydroxyl groups (-OH) is carried out per 1mg, and after shaking 50ml of sodium-hydroxide water solutions of a decinormal for 4 hours and making them react, it filters, 20ml of supernatants of filtrate is extracted, and it titrates in the hydrochloric-acid water solution of the centinormal. From this, the total quantity of the amount of hydroxyl groups and the amount of carboxyl groups was calculated by the bottom type as milliequivalent in carbon black 1g (meq./g).

(Carboxyl group + hydroxyl group) amount = $(50/20 \times 0.01 \times (\text{a titration value} - \text{sky titration value}))$ / carbon black sample Shigekazu -- the amount of carboxyl groups calculated previously was deducted from this total quantity, and the amount of hydroxyl groups was calculated.

(3) Carry out weighing capacity of the measurement desiccation carbon black 3g of the amount of quinone radicals ($>C=O$) per 1mg, and measure the hydrogen capacity generated when the sulfuric-acid water solution of six conventions is added after making the solution which dissolved 100mg of sodium borohydrides in the sodium-hydroxide water solution of a decinormal react with 50ml for 2 hours. The hydrogen capacity which generates by performing a blank test is calculated. The amount of quinones is calculated by the degree type as milliequivalent in carbon black 1g (meq./g).

The amount of quinones = the amount of sodium borohydrides / carbon black sample weight which reacted [Table 1]

カ、ボラ	粒子径	pH	比表面積	表面官能基量
タイプ	(nm)		(m ² /g)	(meq/g)
A	1.2	2.5	460	
B	1.8	8.0	260	
C	1.1	7.0	210	

Carbon black rack A shown in example of manufacture 1 table 1 Zirconia beads were added to the constituent which consists of the 12.0 sections, N, and N-diethylamino propylamine 3.1 section and the toluene 84.9 section, and the paint shaker (red devil company make) distributed for 3 hours. Then, after having carried out the ** exception, having dried at 105 degrees C for 5 hours, performing the Soxhlet extraction of 20 hours by having used THF as the solvent and removing excessive N and N-diethylamino propylamine, it dried and ground at 105 degrees C again for 5 hours, and the surface-preparation carbon black of this invention was obtained.

Except having heat-treated at 70 degrees C after example of manufacture 2 distribution for 24 hours, the same actuation as the example 1 of manufacture was performed, and surface-preparation carbon black was obtained.

Example of manufacture 3 carbon-black rack A Zirconia beads were added to the constituent which consists of the 12.0 sections, N, and N-dimethylamino propylamine 2.5 section and the toluene 85.5 section, the paint shaker (red devil company make) distributed for 3 hours, and it heat-treated at 70 degrees C for 24 hours. Then, the same actuation as the example 1 of manufacture was performed, and surface-preparation carbon black was obtained.

Example of manufacture 4 carbon-black rack B Zirconia beads were added to the constituent which consists of the 12.0 sections, N, and N-diethylamino propylamine 3.1 section and the toluene 84.9 section, the paint shaker (red devil company make) distributed for 3 hours, and it heat-treated at 70 degrees C for 24 hours. Then, the same actuation as the example 1 of manufacture was performed, and surface-preparation carbon black was obtained.

Example of manufacture 5 carbon-black rack C Zirconia beads were added to the constituent which consists of the 12.0 sections, N, and N-diethylamino propylamine 2.5 section and the toluene 85.5 section, the paint shaker (red devil company make) distributed for 3 hours, and it heat-treated at 70 degrees C for 24 hours. Then, the same actuation as the example 1 of manufacture was performed, and surface-preparation carbon black was obtained.

Surface-preparation carbon black was obtained by the same actuation as the example 2 of manufacture except not performing an example of manufacture 6 distribution stroke.

Example of comparison manufacture 1 carbon-black rack A Zirconia beads were added to the constituent which consists of the 12.0 sections, 1, the 3-diaminopropane 1.8 section, and the toluene 86.2 section, the paint shaker (red devil company make) distributed for 3 hours, and it heat-treated at 70 degrees C for 24 hours. Then, the same actuation as the example 1 of manufacture was performed, and surface-preparation carbon black was obtained.

Example of comparison manufacture 2 carbon-black rack A Zirconia beads were added to the constituent which consists of the 12.0 sections, the methylamino propylamine 2.1 section, and the toluene 85.9 section, the paint shaker (red devil company make) distributed for 3 hours, and it heat-treated at 70 degrees C for 24 hours. Then, the same actuation as the example 1 of manufacture was performed, and surface-preparation carbon black was obtained. The surface-preparation carbon black obtained in the above examples 1-6 of manufacture and examples 1-2 of comparison manufacture was shown in Table 2.

[Table 2]

	製造例 比較製造例	全表面 官能基量 (mmol/g)	表面処理剤	分散 力 有無	加熱 温度 (℃)	表面処 理剤の 種類
製造例 1	A	5.1 (9.5)	N,N-ジメチルアミノプロピルアミン	有	—	a-1
製造例 2			N,N-ジエチルアミノプロピルアミン	有	70	a-2
製造例 3			N,N-ジエチルアミノプロピルアミン	有	70	a-3
製造例 4	B	1.1 (7.6)	N,N-ジメチルアミノプロピルアミン	有	70	b-1
製造例 5	C	0.1 (1.9)		有	70	a-4
製造例 6				—	—	a-5
比較製造例 1						
比較製造例 2						

(Examples 1-6) The steel bead was added to the mixture of the following raw material [the surface-preparation carbon black obtained in the examples 1-6 of manufacture, acrylic resin (acid-number 16 mgKOH/g, weight average molecular weight 60000, 50% of solid content), toluene, ethyl acetate, and Solvesso 100], it distributed for 90 minutes using the paint shaker, and carbon black dispersing element

concentration liquid was created.

Surface-preparation carbon black The 10.3 sections Acrylic resin The 25.1 sections Toluene The 32.3 sections Ethyl acetate The 12.9 sections Solvesso 100 Raw material of the above-mentioned dispersing element concentration liquid and the following in 19.4 ***** (what has acrylic resin the same as the above, you van 20SE60: melamine resin made from Mitsui SAITEKKU) It mixed and acrylic resin / melamine resin system coating was obtained.

The above-mentioned dispersing element concentration liquid The 35.4 sections Acrylic resin The 49.9 sections n-butanol The 2.5 sections You van 20SE60 Set to the coating constituent of the 12.2 section (examples 1-5 of comparison) examples 1-6. Acrylic resin / melamine resin system coating was obtained like examples 1-6 except having changed the used surface-preparation carbon black into the surface-preparation carbon black of the examples 1-2 of comparison manufacture, and unsettled carbon black A, B, and C. Evaluation of the coating created in the above examples 1-6 and the examples 1-5 of a comparison was performed as follows. After having painted the coating by the bar coating machine so that the thickness of a dry paint film might be set to about 30 micrometers, and putting it on a PET film for 30 minutes, 140 degrees C could be burned for 30 minutes, the paint film was created, and gloss (60-degree-C mirror reflectivity), reflection density, and Japanese lacquer malanism were evaluated.

Moreover, in the base coatings of titanium oxide, the coating was cut so that the ratio of surface-preparation carbon black and titanium oxide might become 1/10, the light color coating was adjusted, drawdown and after putting for 30 minutes, it could be burned in the white paper for 30 minutes at 140 degrees C by the 6-mil film applicator, and the paint film was created, and tinting strength was evaluated. In addition, various kinds of test methods are as follows.

[gloss (60-degree mirror reflectivity)]: glossmeter [-- Suga Test Instruments Co., Ltd.: -- the 60 degree-60" reflection factor of a paint film was measured by digital deflection glossmeter UGV-5D].

[reflection-density]: Macbeth densimeter [Macbeth The reflection density of a paint film was measured by RD-918].

[Japanese-lacquer malanism]: Viewing estimated the blackness of a paint film.

O : -- very much -- fitness **: -- a little -- fitness x: poor [tinting-strength]: colorimeter [-- Suga Test Instruments Co., Ltd. SM color computer SM-7] -- lightness-index difference with the example 4 of a comparison L was measured and it asked for tinting strength from the degree type. Dispersibility is so good that this numeric value is large.

Tinting strength = $100 - (L \times 10)$

These test results were shown in Table 3.

[Table 3]

	实施例	分散性			
		分散性	分散性	分散性	分散性
		分散性	分散性	分散性	分散性
实施例 1	a-1	8.8	分散性		分散性
实施例 2	a-2	9.1	分散性		分散性
实施例 3	a-3	9.2	分散性		分散性
实施例 4	b-1	9.4	分散性		分散性
实施例 5	c-1	8.9	分散性		分散性
实施例 6	a-4	8.0	分散性		分散性
比较例 1	a-5	7.7	分散性		分散性
比较例 2	a-6	8.0	分散性		分散性
比较例 3	A	7.8	分散性		分散性
比较例 4	B	7.3	分散性		分散性
比较例 5	C	7.1	分散性		分散性

From Table 3, the coating using the surface-preparation carbon black obtained from this invention is understood that dispersibility is improving remarkably.

[Translation done.]